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Microscale Preparation of Pyrocatechol: The Use of Sodium Percarbonate in the Dakin Reaction

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Sodium percarbonate ($\text{Na}_2\text{CO}_3 \cdot 3/2\text{H}_2\text{O}_2$), a common household detergent, is an inexpensive, stable, and easily handled reagent that has an excellent shelf life. One equivalent of sodium percarbonate releases one and one-half equivalents of hydrogen peroxide. Sodium percarbonate (Fig. 1) has been used for the oxidation of sulfides (Ando, Chem. Lett., 665, 1986), amines (Zazac, J. Org. Chem., 54:2468, 1988), and organoboranes (Kabalka, Organometallics, 9:1316, 1990). We wish to report the use of this reagent for the preparation of pyrocatechol from salicylaldehyde (Yamazaki, M.S. Thesis, University of Arkansas at Little Rock, 1991), a Baeyer-Villiger type reaction commonly referred to as the Dakin reaction (Dakin, Org. Synth., Coll. Vol. 1: 149-150, 1964).

hydroxyacetophenone in 69% yield. In closely related experiments, hydroquinone was prepared from 4-hydroxybenzaldehyde in 35% yield as well as from 4-hydroxyacetophenone in 66% yield (Evans, M.S. Thesis, University of Arkansas at Little Rock, 1991). In all cases the ir and nmr spectra of the products were identical to those of authentic samples.

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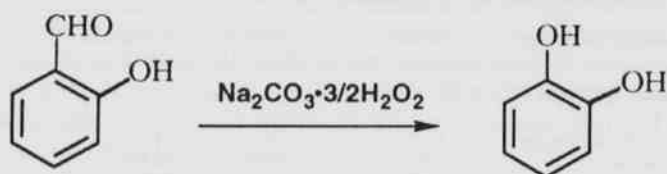


Fig. 1. Sodium percarbonate

To a solution of salicylaldehyde (244 mg, 2 mmol) in 3 ml of 1 N sodium hydroxide was added gradually sodium percarbonate (630 mg, 6 mmol). A vigorous exothermic reaction occurred immediately with darkening of the mixture which was then stirred for 24 hours in a sandbath at 50°C. After chilling the reaction mixture in an ice-bath and neutralization with acetic acid, the solvent was completely removed under reduced pressure leaving a dry black residue. The residue was finely crushed and extracted with toluene overnight using a microscale Soxhlet extraction apparatus. The toluene solution was evaporated to yield crude pyrocatechol which was further recrystallized from toluene to afford 0.11 g of pure compound in 50% yield, mp 102-104°C (Lit. 105°C).

Similarly, pyrocatechol was also obtained from 2-